Please add claims 8 and 9 as follows.

8. A process as in claim 1, wherein the adsorption, deposition, and washing steps are repeatedly conducted for at least 4 cycles.

9. A process as in claim 8, wherein the adsorption, deposition, and washing steps are repeatedly conducted for a number of cycles in the range of from 4-9.

REMARKS

The application has been amended. In particular, claim 1 has been amended to include specific limitations regarding the deposition of the metal extending into the interior of the ion-exchange resin product near the surface. Support for this amendment can be found in the application as filed on page 20, lines 12 through 15. Also, new dependent claims 8 and 9 have been added, which include subject matter previously set forth in claim 1. As such, the amendments are not considered new matter within the meaning of 35 U.S.C. § 132. In view of the amendments above and the remarks below, reconsideration is respectfully requested.

Claim 1 has been rejected under 35 U.S.C. § 103(a) as being unpatentable over PCT Publication No. WO 97/26039 to Shahinpoor et al. (hereinafter "Shahinpoor") alone or in combination with U.S. Patent No. 4,364,803 to Nidola et al. (hereinafter "Nidola"). This rejection is respectfully traversed.

As the Examiner is aware, the present invention is directed to a process for producing an actuator element involving forming metal electrodes on an ion-exchange resin product through repeatedly conducting the sequential steps of adsorption, deposition, and washing. By repeatedly conducting these steps, metal deposition further proceeds to the interior of the ion-exchange resin product, as set forth in the amended claim. Such progress of deposition of the metal to the interior surface of the resin product increases the contact area between the ion-exchange resin product and the metal electrode which increases the quantity of ions migrating

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to the electrode. Such a process reduces the surface of resistance of the electrode and improves the conductivity thereof, and further results in a high degree of bending, therefore providing quick response for the electrode in its intended use as an actuator element.

The Shahinpoor and Nidola references, whether considered alone or in combination, fail to disclose repeatedly conducting the adsorption, deposition, and washing steps, let alone the progress of such steps to cause deposition of the metal in the interior of the ion-exchange resin product, as set forth in the present claims. Moreover, nothing in either of these references discloses that improved bending or deformation can be achieved through such repeated processing steps causing deposition to proceed to the interior of the resin product.

Shahinpoor merely discloses a method for creating an actuator through reduction on an ion-exchange material. In Shahinpoor, a coating and a reducing step are disclosed. Shahinpoor further discloses that the reducing and rinsing steps can be repeated. Nothing in the Shahinpoor reference, however, discloses repeatedly conducting an adsorption step, a deposition step, and a washing step sequentially to deposit the metal not only on the surface of the ion-exchange resin product but also in the interior near the surface.

In fact, Shahinpoor notes at page 13, lines 6-8 that "the amount of platinum deposited on the membrane must be about 3.75 mg/sq cm for each face of the membrane surface. This produces a pair of porous dendritic electrodes on the surface of the membrane." As such, Shahinpoor merely teaches deposition on the surface of the membrane and fails to describe deposition of the metal electrode in the interior of the ion-exchange resin product near the surface. Such deposition on the interior of the ion-exchange resin product results in the quantity of ions migrating to the electrode being increased, as discussed above. Such deposition further results in reduced surface resistance of the electrodes, and improved conductivity thereof. Such results are not suggested in the Shahinpoor reference.

The Examiner has cited the Nidola reference, contending that Nidola teaches controlling the amount of metal deposited on the surface of ion-exchange resins during the reductive deposition by repeating the step of adsorbing metal complexes. The Examiner then concludes that it would have been obvious to repeat the step of adsorbing a metal complex in the Shahinpoor reference, because Nidola teaches the embodiment of repeating the metal complex adsorption step. The Examiner concludes that such a combination renders the present invention obvious.

Nidola discloses a uniform metal coating formed on the surface of a polymeric ion-exchange resin membrane by chemical reduction of a metal ion adsorbed on the surface of the membrane. The metal electrode in Nidola is formed on the surface of the membrane. Nothing in the Nidola reference teaches or suggests that the metal electrode is deposited in the interior of the membrane near the surface. Accordingly, Nidola fails to add any teachings to the deficiencies of Shahinpoor to render the present claims obvious, which clearly require repeatedly conducting the adsorption, deposition, and washing steps to deposit the metal on the surface of the ion-exchange resin product as well as in the interior of the ion-exchange resin product.

In fact, Nidola notes that at column 5, lines 13-14 that the adsorption of the metal salt before contact with the reducing agent makes it possible to obtain better adherence between the metal film and the membrane, and at column 8, lines 42-43 notes that a negligible electric resistivity between two laterally spaced points on the coating is obtained by repeating the operations 2-4 times. Nidola merely aims to improve adherence and lower resistivity of the coatings. On the other hand, by depositing the metal on the interior of the ion-exchange resin product, the contact area is increased, and the quantities of ions migrating to the electrode are increased. As such, a high degree of bending and displacement is exhibited. Neither Nidola nor Shahinpoor, considered alone or in combination, disclose or even remotely suggest that such

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effects can be seen through repeatedly conducting the sequence of adsorption, deposition, and washing.

In fact, the specification clearly sets forth that optimization of the sequence of adsorption, deposition, and washing is achieved by conducting such a sequence for a cycle of from 4-9 times. Attached to the present amendment is a copy of a paper entitled "Morphology of Electrodes and Bending Response of the Polymer Electrolyte Actuator", as presented by the inventors of the present application. *Electrochimica Acta 46 (2000) 737-743.* The results set forth in this *Electrochimica* article clearly demonstrate that repeatedly conducting the sequence of adsorption, deposition, and washing increases the deposition of the metal to the interior of the ion-exchange resin product, and leads to improved displacement. For example, Fig. 8 of this article depicts SEM micorgraphs, which clearly shows the improved deposition within the interior structure of the ion-exchange resin product over repeated plating cycles. Moreover, Fig. 8 of the article shows peak-to-peak displacement of the composite membrane driven by a two-V square wave versus the number of plating steps. As seen through Fig. 8, the displacement of a sample having one plating cycle is lower than 0.1 mm, while those of two or more plating cycles are higher than 0.5 mm. Clearly, repeatedly conducting the sequencing cycles leads to the samples having higher displacement.

The sequential plating involving repeating the steps of adsorption, reduction, and washing greatly improves the actuator displacement by build up of the metal on the interior of the ion-exchange resin product. The results further demonstrate that an optimum number of plating cycles for such sequential cycling is demonstrated, beyond which little improvement in displacement occurs. Nothing in the teachings of Nidola nor Shahinpoor disclose or suggest deposition through such sequential plating to achieve deposition at the interior of an ion-exchange resin product, let alone optimization of such sequential cycling to achieve improvements in displacement. In fact, nothing in either of these references discusses the

possibility of improved displacement through the sequential cycling of the plating steps. Withdrawal of the rejections based on these references therefore is respectfully requested.
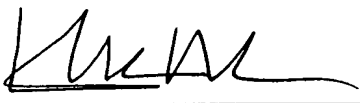
Applicants note that the paper attached hereto demonstrates the improved results through repeatedly conducting the adsorption, deposition, and washing steps of the present invention. Applicants can present such evidence in the form of a Declaration if required by the Examiner.

In view of the above remarks, reconsideration and withdrawal of the rejection are respectfully solicited. Applicants also respectfully request that any further action by the Examiner be proceeded by a telephone call to Applicants' undersigned representative to expedite prosecution of the present application.

Should the Examiner have any questions regarding any of this information, the Examiner is invited to contact Applicants' undersigned representative by telephone at (412) 471-8815.

Respectfully submitted,

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1.   (Thrice Amended)   A process for producing a polymeric actuator comprising an ion-exchange resin product and metal electrodes which are formed on the surface of the ion-exchange resin product and are insulated from each other, said actuator operating as an actuator by applying a potential difference between the metal electrodes when the ion-exchange resin product is in the water-containing state to allow the ion-exchange resin product to undergo bending or deformation,

wherein the following steps (i) to (iii) are repeatedly conducted to form the metal electrodes ranging from the surface of the ion-exchange resin product to the inside thereof;

(i)   a step of allowing the ion-exchange resin product to adsorb a metal complex in an aqueous solution (adsorption step),

(ii)   a step of reducing the metal complex adsorbed on the ion-exchange resin product by a reducing agent to deposit a metal on the surface of the ion-exchange resin product (deposition step), and

(iii)   a step of washing the ion-exchange resin product having the deposited metal (washing step),

[the number of cycles of the above steps is in the range of 4 to 9; wherein

in the case of an ion-exchange resin product being a plate or a film, the ratio of the thickness (a1) of the metal electrode formed on the ion-exchange resin product to the thickness (b1) of the ion-exchange resin product including the metal electrode (a1/b1) is in the range of 0.03 to 0.40,

in the case of the ion-exchange resin product being a cylinder with the metal electrode formed on an outer or inner surface thereof, the ratio of the thickness (a2) of the metal

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electrode formed on the ion-exchange resin product to the thickness (b2) of the cylindrical ion-exchange resin product including the metal electrode (a2/b2) is in the range of 0.02 to 0.70,

in the case of the ion-exchange resin product being a cylinder with the metal electrode formed on both an inner and outer surface thereof, the ratio of the thickness (C) of the cylindrical ion-exchange resin product excluding the metal electrode to the thickness (b4) of the cylindrical ion-exchange resin product including the metal electrode (C/b4) is in the range of 0.20 to 0.95.] ,such that the deposition of the metal is conducted not only on the surface of the ion-exchange resin product but also in the interior near the surface.

Morphology of electrodes and bending response of the polymer electrolyte actuator

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Abstract

In order to induce bending motion in a perfluorinated polymer electrolyte by electric stimuli in water or saline solution, plating with metal is required. To fabricate electrodes, a perfluorocarboxylic acid membrane was soaked in Au(III) di-chloro phenanthroline complex solution, and then any adsorbed Au(III) cation complex was reduced in aqueous sodium sulfite. Optimizing the motion response depends on control of the chemical plating procedure. By sequential adsorption/reduction cycling, a suitable pair of gold electrodes with a fractal-like structure have been grown. This high interfacial area between the electrodes and polymer electrolyte leads to larger deformation. The measured deformation progressively improves with cycling. Displacement rates were proportional to current. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Polymer electrolyte; Gold electrode; Polymer–metal composite; Micro machine; Soft actuator

1. Introduction

Micro-electro-mechanical devices can be made with current IC technology [1]. However, the technology is complex and expensive. Recently, considerable attention has been paid to actuators based on the deformation of polymer gels in response to environmental change [2]. These so-called mechano-chemical actuators convert chemical, thermal or photon energies into mechanical energy due to polarity change. These devices are simple and easy to design and fabricate. Soft actuator based on electro conductive polymer has been developed by several workers [3–5]. Conductive polymers such as polyaniline, polypyrrole, and polyacetylene can increase their electrical conductivity from the insulator to metallic regime upon the occurrence of certain chemical or electrochemical doping reactions. These reactions have been shown to be accompanied by a change in the volume of the polymer, such that the polymers can be manipulated to expand or contract.

Advances have been made with polymer electrolyte membranes plated with platinum, which deform under electric stimuli in water or saline solution [6–8]. These polymer electrolyte actuators (PEAs) have superior deformation, and quick response, but normally require substantial applied voltage to give sufficient deformation, and introduce electrolytic effects, e.g. gas production.

In the field of medical equipment and micro-robotics, there is great demand for a miniaturized, lightweight and flexible actuator. PEAs are particularly suitable for in vivo applications provided electrolysis is eliminated. The polymer electrolyte actuator effectively comprises a solid polymer electrolyte, at least two electrodes, coun-

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ter ions (e.g. H^+, Na^+), and any solvent present in-situ. The response of the PEA is controlled by many factors; not only the intrinsic properties of its components, but also the dimensions of the device, and details of the electrode-forming process.

Perfluorinated polymer electrolyte membranes in the carboxylic acid form are superior to the corresponding sulfonic acid form for this application. They have higher ion-exchange capacity and better mechanical strength. Gold is a promising material as electrode, being stable in acid, yet softer, more conductive, and less active in electrochemical reactions than platinum [9,10].

Previous papers [11,12] have proposed the deformation model of the PFAs, in which the deformation is attributed to the osmotic drag water flow associated with the capacitive current. The model successfully predicted the bending behavior of the solid polymer electrolyte membrane platinum composite, quantitatively. According to the model proposed, the amplitude of the deformation is proportional to the capacitive current and a transference coefficient of water. Hence, a high interfacial area between the electrodes and polymer electrolyte should lead to larger deformation [13,14]. Other factors conducting to good response are high electrode conductivity and efficient water transport as solvation sphere of the migrating cation, which ideally is highly hydrophilic, i.e. having a high 'osmotic drag' coefficient.

This report illustrates the advantage of optimizing the interfacial area between electrode and membrane to enhance deformation response with minimal electrolysis. To achieve this, gold deposits in the film are accumulated by sequential adsorption–reduction plating cycles. The morphology of the deposited electrode and the factors affecting the deformation performance are discussed.

2. Experimental

2.1. Preparation of the composite

Fig. 1 is a schematic representation of method for preparation of a gold–Flemion® composite. The Flemion® (Asahi Glass Co., Ltd.) is perfluoro carboxylic acid cation-exchange membrane with an ion-exchange capacity of 1.44 meq/g and 0.14 mm thick. After surface roughening by 'dry-blasting', both sides of the membrane, were chemically plated with gold as follows [9,10]. The perfluorocarboxylic acid membrane was soaked in Au(III) di-chloro phenanthroline complex solution for > 10 h at room temperature, rinsed, and then any adsorbed Au(III) cation complex in the membrane was reduced in aqueous sodium sulfite at 40–70°C for 6 h.

By sequential adsorption–reduction cycling up to 8 times, a suitable gold loading in the membrane may be accumulated. After washing the composite membrane in 4N HCl at 70°C, the membrane was immersed in 0.1N NaOH at room temperature for > 12 h to exchange to the sodium (Na^+) form.

2.2. Characterization of the composite membrane

The thickness of the gold layer electrode was measured by a fluorescent X-ray coating thickness gauge of SEIKO Industrial & Electronics, which has a dynamic response up to 5 μm. The surface resistance of the plated electrode was measured with a Kaise SK-6155 multi-meter. The composite was then cut into a ribbon with a width of 1 mm, and the resistance measured across a length of 7 mm. The interfacial area was evaluated by electric double layer capacitance determined from cyclic voltammetry (− 0.5 to + 0.5V) without reference electrode at a scan rate of 10 mV s⁻¹ scan
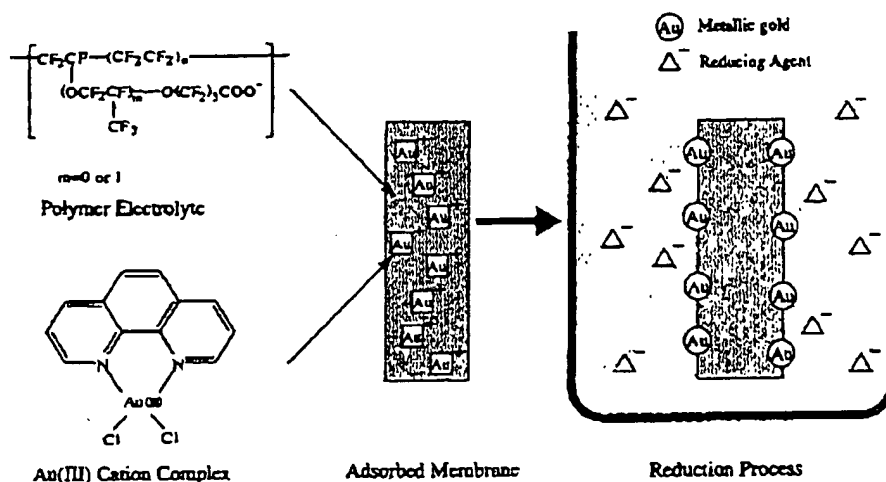

Fig. 1. Schematic representation of gold–solid polymer electrolyte composite prepared by adsorption–reduction method.
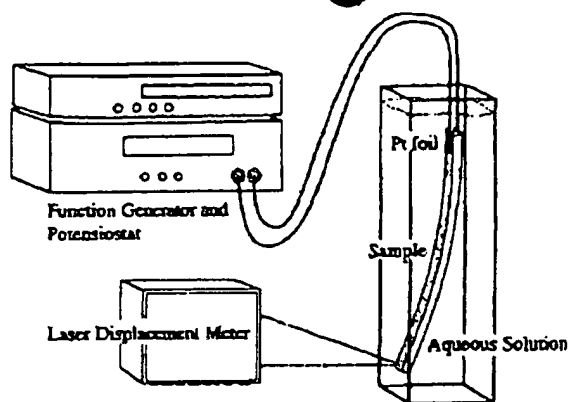
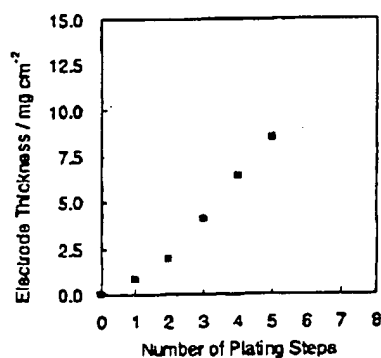Fig. 2. A schematic of displacement measuring system.

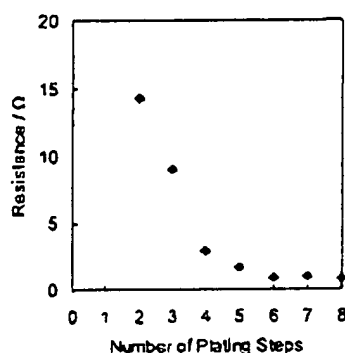Fig. 3. Electrode thickness of a single side in the membrane.

Fig. 4. Electrical resistance of the membrane surface ($L = 7$ mm) (sample size: 1.0×7.0 mm).

rate (Electrochemical Workstation BAS 100 B/W, Bio-analytical Systems, Inc.). The distribution of gold deposits in the membrane and their cross-sectional morphology were examined with scanning electron microscopy at 15 KV acceleration voltage (Hitachi SEM S-2500).

2.3. Deformation testing

Fig. 2 is a schematic of the set-up for evaluating bending response. The plated polymer electrolyte membrane was cut into a ribbon of 1 mm width and 8 mm length. The ribbon was supported vertically in water by clamping one end in platinum foil to make electrical contact with the gold electrode. Electric signals were supplied from a Yokogawa AG1200 waveform generator and a Hokuto Denko HA-501G potentiostat/galvanostat. Displacement of the free end at a distance of 5 mm from the fixed point was measured by a Keyence LC-2220/2100 laser displacement meter directed through the glass sample cell at room temperature.

The displacement response was stimulated by a square-wave and triangular-wave input signal (± 2.0 V) at a frequency of 0.1 Hz.

3. Results

3.1. Characterization of the composite membrane

3.1.1. Electrode thickness

Fig. 3 shows the development of electrode thickness in the plated membrane. By adsorption–reduction cycling, the thickness increases continuously with the number of plating steps, though it appears that the ion exchange capacity is unaffected by progressive accumulation of Au deposits.

3.1.2. Surface resistance of electrode

Fig. 4 shows the drop in surface resistance of the electrode with plating sequence. A working value of $< 3 \, \Omega$ (sample size: 1×7 mm) was typically reached after 4 plating cycles.

3.1.3. Interfacial area between ion exchange polymer and the electrode deposit

Fig. 5 shows SEM micrographs of the cross section of the composite membrane. By adsorption–reduction cycling, a fractal-like structure of gold with high interfacial area within the membrane is obtained. The membrane structure depends on the conditions of plating and surface-roughening pre-treatment. The distribution of gold in the membrane is very sensitive to the reduction condition.

The interfacial area can be evaluated because it is proportional to the electric double-layer capacitance, which can be measured easily. Fig. 6 shows a representative cyclic voltammogram of the membrane in water. Electrode potential was cycled within the double-layer charging region (-0.5 to $+0.5$ V), and the scan rate was 10 mV s^{-1}. In the absence of electrochemistry, the double-layer capacitance is the ratio of current to voltage scan rate. Fig. 7 shows the trend in the capaci-
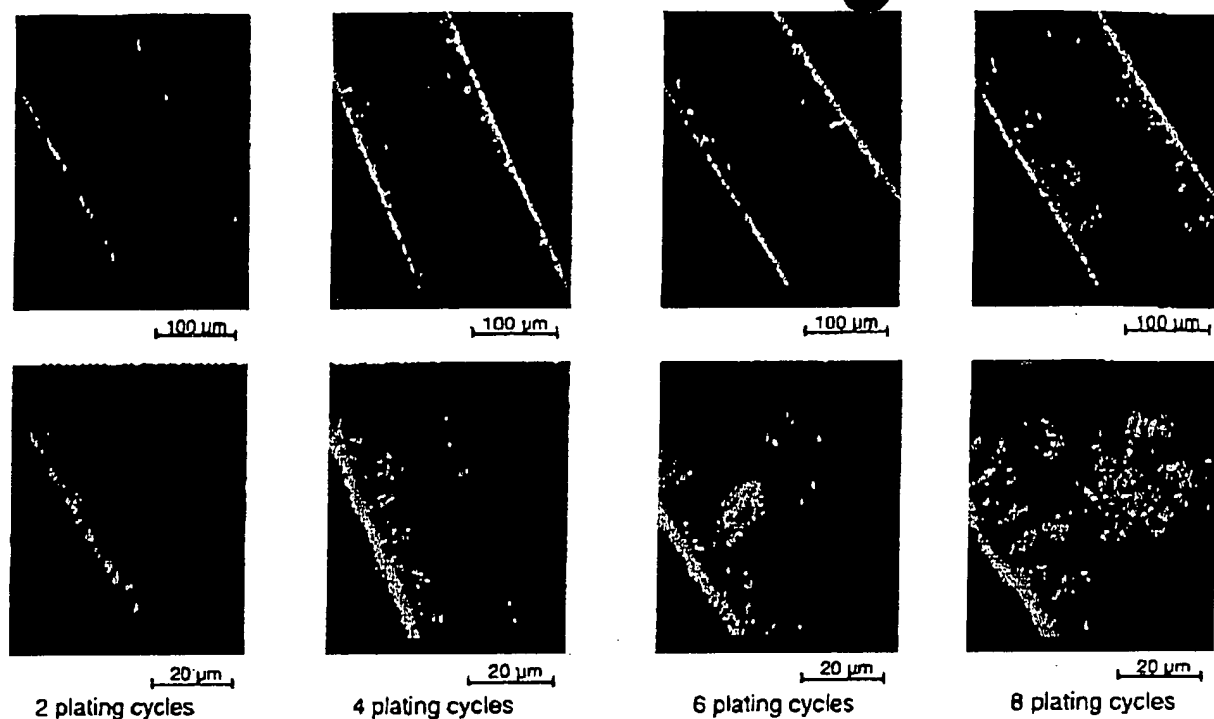
| 2 plating cycles | 4 plating cycles | 6 plating cycles | 8 plating cycles |

Fig. 5. SEM micrograghs in cross-section of membrane electrode.

tance of the membrane with plating cycle. To check reproducibility, the data for two samples is illustrated. Both show a systematic increase in capacitance up to ~6 or 7 plating cycles but with a clear approach to a limiting value or saturation.

3.2. Analysis of the deformation

Fig. 8 shows the effect of plating cycles on peak-to-peak displacement of the composite membrane (Na^+-form) on applying a 2 V square wave at 0.1 Hz. The displacement increased with the number of plating cycles up to roughly 6 times, but showed no clear im-

provement beyond. The interfacial area begins to decrease and/or the hardness of the electrode increases with excessive plating, thus becoming deleterious for performance.

Fig. 9 shows the development of displacement with electric double layer capacitance. The capacitance increased the displacement proportionally except plot of 462 μF cm^{-2}. The displacement at the plot was given low value caused of high resistance with electrodes.

Fig. 10 shows the waveforms of current and displacement response of the composite membrane (Na^+ - form) at 2 V applied as a triangular wave-form at 0.1 Hz. At region A, some kind of redox current and the increase in the slope of the displacement was observed. At other regions during the sweep of voltage, the

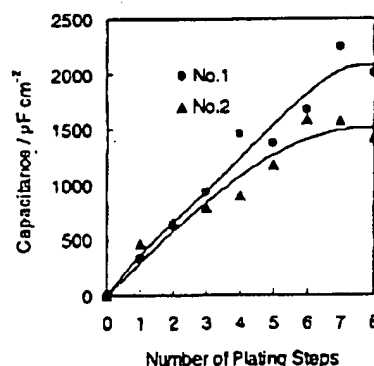Fig. 6. Cyclic voltammogram of the composite in water.

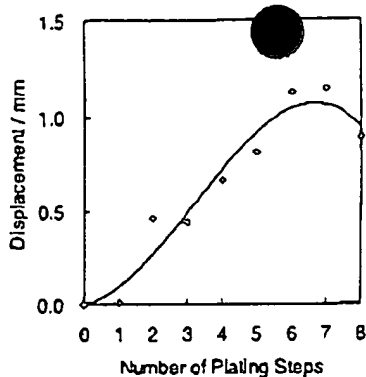Fig. 7. Electric double layer capacitance.

Fig. 8. Peak to peak displacement of the composite membrane driven by 2 V square-wave vs. number of plating steps.
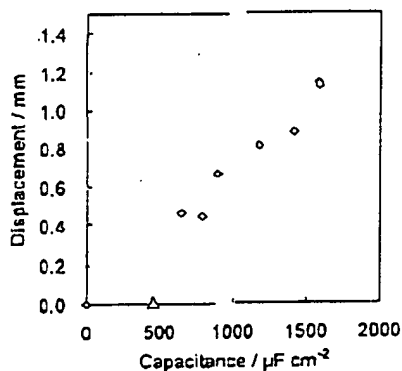

Fig. 9. Displacement vs. electric double layer.

capacitive current was only observed and the displacement linearly increased with time.

Fig. 11 shows the trend in displacement rate with increasing current at point B, as described in Fig. 10.
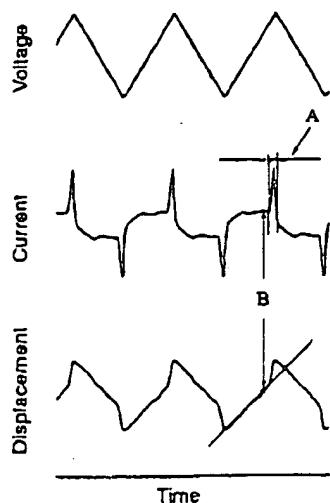

Fig. 10. Waveforms of voltage, current and displacement of membrane. A: electrolysis range; B: maximum current value with no-electrolysis.

After the first platin̶g̶ (̶=̶ 1), It is impossible to measure displacement rate and current because the resistance of the electrode surface is still extremely high.

It is clearly seen that displacement rate is proportional to current, i.e. $d = kI$, where d = displacement rate and k is a proportionality constant. This result is in conformity with the response model proposed in the previous paper. It can also be seen that k progressively drops with plating cycle number (n). Fig. 12 shows that the drop in k is actually proportional to n. Thus, there is a trade-off between amplitude of response (see Fig. 8) and speed of response (Fig. 11) at a given applied voltage and plating degree. The likely factor working against increasing displacement with plating cycle is a concomitant increase in rigidity of the composite with accumulating Au deposition. This result can also be explained by the previously proposed response model.

4. Discussion

The purpose of the sequential plating process presented in this paper is to explore electrode-forming processes of the PEAs with larger deformation. Increasing the number of plating steps results in a decrease in the surface resistance R_s, an increases in the double layer capacitance C_d, and an increase in the rigidity of the composite, k. The value of R_s decreases, sharply, by five plating cycles, and after that, it remains almost constant ($R_s = 1\ \Omega$). Deformation for the composites of 1 plating cycles was not observed, because of the high surface resistivity. Hence, the plating electrode requires more than 4 chemical plating cycles in order to significantly affect surface resistance.

Regarding the rigidity of the composite, the increase in the plating cycles is a disadvantage for larger deformations (Fig. 12). The increase in the double layer capacitance means an increase in the capacitive current. Growing the fractal-like structure of the deposited electrode by the sequential plating as shown in Fig. 5 is important for increasing the values of the double layer capacitance. The large increase in the capacitive current by the growth of the structure leads to larger deformation as the plating cycles, compensating for the negative effect of the rigidity of the composite. However, at eight plating cycles, the fractal-like structure has never been grown, and the double layer capacitance is almost the same as that of seven plating cycles (Fig. 7). Hence, the deformation performance of the composite of eight plating cycles is worse than that of 7 plating cycles as shown in Fig. 8, because of the rigidity of the composite. In consideration of all factors mentioned above, more than 4 plating cycles are needed and six–seven plating cycles are optimum for largest deformation of
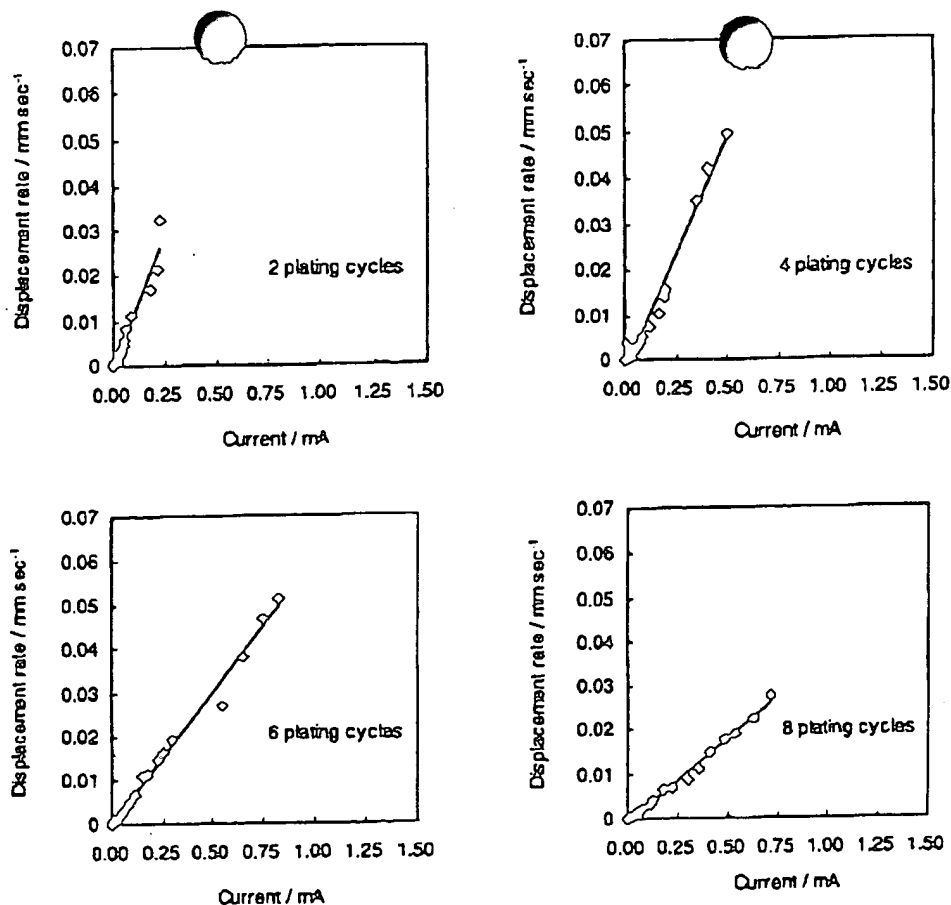
Fig. 11. Displacement rate vs. current.

the PFAs based on the perfluorocarboxylic acid membrane and Au composite.

The methods of forming solid polymer electrolyte membranes (SPM)/metal composites have been originally developed for using the composites as water electrolysis processes [15]. Two different plating methods, known as 'reductant permeation' (RP) [16] and 'impregnation-reduction' (IR) [17], have been developed

Fig. 12. Proportional coefficient vs. number of plating cycles.

for direct contacting of a polymer electrolyte membrane and a metal electrodes under wet conditions. For optimum performance of the PFAs, the metal-plating step should satisfy several criteria, namely, a low surface resistance of plated electrode, a high interfacial area, a wide potential window, and a low rigidity. Some properties of the plated electrode for the water-electrolysis do not meet the criteria. The sequential plating of gold presented in this paper is one of the answers to this problem.

In the near future, the effect of counter ion (Li$^+$, Na$^+$, K$^+$) must be investigated as an important part of the method optimization.

5. Conclusion

In electrode development, a sequential plating (adsorption-reduction) procedure has been shown to greatly improve the actuator displacement by build-up of fractal-like gold deposits which provide a high electrode/membrane interfacial area. The progressive deposition of high specific area improves the absolute
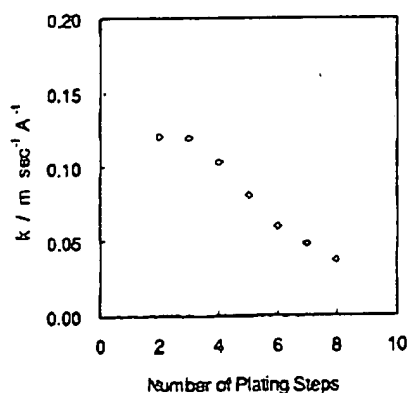
displacement with electrical stimulus over many plating cycles. The plating method must be improved by increasing the surface-to-volume ratio, or 'dispersion', of Au particles. The speed of response is proportional to the induced current, which is in turn a systematic function of plating cycle number. However, an optimum number of plating cycles (~ 6–7) is reached, beyond which little improvement in displacement occurs. Together with a gradual loss in speed of response, these effects suggest a deleterious increase in mechanical resistance or stiffness with excessive accumulation of gold.

Acknowledgements

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